



# The influence of spent household batteries to the organic fraction of municipal solid wastes during composting

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## ARTICLE INFO

### Article history:

Received 18 December 2010

Received in revised form 22 February 2011

Accepted 25 February 2011

Available online 13 April 2011

### Keywords:

Municipal solid wastes

Composting

Household batteries

Heavy metals

## ABSTRACT

The objective of this work was to investigate the potential transfer of 9 heavy metals from spent household batteries (zinc–carbon and alkaline–manganese batteries) to the organic fraction of municipal solid wastes during active composting. Six runs were performed including one control and 2 replications. Eleven types of alkaline and non-alkaline batteries were added at 3 different levels to the organic fraction of municipal solid wastes, namely at percentages equal to 0.98% w/w (low), 5.2% w/w (medium) and 10.6% w/w (high). Experiments were performed in 230 l insulated plastic aerobic bioreactors under a dynamic air flow regime for up to 60 days. Iron, copper and nickel masses contained in the organic fraction of the wastes were found significantly higher in the high level runs compared to the corresponding masses in the control. No metal transfer was obtained in the low and medium level runs. Metal mass balance closures ranged from 51% to 176%. Metals' concentrations in the leachates were below  $10 \text{ mg l}^{-1}$  for most metals, except iron, while an increasing concentration trend versus time was measured in the leachates of the high level runs. In all cases, the contents of 5 regulated heavy metals in all end products were below the Hellenic limits.

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## 1. Introduction

Household batteries are common constituents of municipal solid wastes. Recently, there have been major efforts in Europe to separate spent household batteries (SHB) from the municipal solid waste (MSW) stream in order to recycle them. According to the European legislation (EC, 2006) 25% of the batteries produced in the European member states must be collected and recycled in 2012; 45% must be recycled in 2016. In Greece, the separate collection of small household batteries started in 2004 (AFIS S.A., 2010). According to AFIS S.A. (2010), 629 t of SHB were collected and recycled in 2009 in Greece, which was an increase of 27% compared to 2008. The amounts collected in 2009 were 31% of the total production of batteries in 2009. Still, significant amounts of SHB apparently end up to the commingled municipal solid waste stream.

Household batteries fall under under code number 16.06 of the European Waste Catalogue (EC, 2000). They are designated as *non-hazardous wastes*, unless they are Ni–Cd type or lead type batteries or they contain mercury. In the latter cases, batteries are designated as *hazardous wastes*.

The heavy metal content of MSW could be partially attributed to the presence of batteries although no clear data exist to specify the magnitude of influence. The composition of spent household batteries has been occasionally studied by researchers (Panero et al., 1995; Bartolozzi et al.,

1994; Almeida et al., 2006; Richter et al., 2008; Recknagel et al., 2009). Panero et al. (1995) were among the first to measure total metal contents in selected household batteries; they found that Zn and Mn were the dominant metals followed by lead, and cadmium in traces, while Hg was absent. In the work of Recknagel et al. (2009), all types of batteries had a Hg content lower than  $3.9 \text{ mg kg}^{-1}$ . Lead and cadmium were much higher in zinc–carbon batteries compared to the alkaline–manganese ones. Still all batteries met the heavy metal requirements in Germany. In Richter et al. (2008), the zinc–air button cells also met the German requirements for heavy metal content.

As of today, there is limited information on the effect of SHB to MSW treatment and disposal processes. Emphasis has been mostly given to the quality of leachates using leaching tests or after simulating anaerobic landfill environments. Panero et al. (1995) measured the metal concentration in simulated acidic leachates and they found that concentrations of Ni, Zn, Cd and Mn were, each, lower than  $2 \text{ mg l}^{-1}$ . Selvapathy and Madhavan (2003) performed Toxicity Characteristic Leaching Procedures (TCLP) on a wide range of battery types. According to their results, zinc–carbon and alkaline batteries highly exceeded the regulatory TCLP limits for zinc and manganese. The Ni–Cd batteries, on the other hand, exceeded the cadmium and nickel regulatory limits. No mercury was detected in their analyses. Xará et al. (2009) performed four standardized leaching tests in alkaline household batteries and they found significant differences in the results of the 4 tests; still, zinc was the dominant metal leached, followed by lead, nickel and copper. Karnchanawong and Limpitprakan (2009) performed leaching tests on several batteries; in addition, they were the first to use anaerobic lysimeters to simulate landfill environments and measure the leachability

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of batteries in such environments. They added batteries at percentages equal to 1% and 5% to simulated MSW in the anaerobic lysimeters. The authors found that zinc and manganese were the dominant metals leached followed by nickel, cadmium and lead. As the pH of the leachate decreased in the MSW mixtures during the acidogenic phase, metals—particularly lead and mercury—became more soluble. In a recent work, Almeida et al. (2009) investigated the effect of spent household batteries on emissions during MSW incineration; zinc and mercury were found to be the most volatile metals.

There seems to be no information on the behavior of SHB during a MSW composting process. Such information could aid in determining the extent of the removal of SHB from the MSW stream prior to a composting treatment facility. This lack of information led to the current research that aims to answer the question: “Do heavy metals transfer from the batteries to the compostable product during an active composting period, and if yes, to what extent?”. Based on the above, selected zinc–carbon and alkaline–manganese household batteries were added to the organic fraction of MSW (OFMSW) at 3 different concentrations. Experiments were performed in pilot scale aerobic bioreactors with a simulated OFMSW that contained food wastes and office paper. The experiment simulated an active composting process that lasted up to 60 days. The curing process—which may last up to several months—was not simulated in this experiment. Therefore, the experiment was designed on the notion that any batteries present in an MSW stream directed to a composting facility, will remain there, during the active composting stage, but will be removed prior to curing via screening. Screening, after the active composting process, and prior to curing, is anyways a common practice in MSW field composting facilities (Lau et al., 2005).

In this research work, the simulated OFMSW was spiked with SHB at 3 concentration levels. Objectives of the work were to calculate statistically significant variations among metal masses during the composting process and to perform a mass balance of the metals at the start and end of the composting process. That is, the research aimed to answer the question of whether any metals transfer from the batteries to the OFMSW during the active composting stage.

## 2. Materials and methods

### 2.1. Simulated organic fraction of MSW

The organic fraction of MSW (OFMSW) was simulated using food wastes (FW) and wasted office paper (OFP); these were mixed at an

approximate ratio of 70%:30% (FW:OFP) on an as discarded (wet) weight basis (ww). This mixing ratio is based on the typical Hellenic MSW composition. The above mixing ratio corresponds to a ratio of 47%:53% (FW:OFP) on a dry weight basis (dw) and was consistently maintained throughout all runs. A fifty (50) kg (ww) batch of food waste was collected from the university restaurant over a week. The food wastes comprised of cooked meat, bread, vegetables, fruit and cooked pasta. The food waste batch was thoroughly mixed with a shovel and was stored at a  $-20^{\circ}\text{C}$  temperature for use throughout the experiments. Food waste was left to thaw for at least 8 h, prior to its introduction into the bioreactors. The wasted office paper was collected from faculty offices and was shredded manually in pieces of approximate dimensions of  $2.5\text{ cm} \times 2.5\text{ cm}$ . Both materials (FW and OFP) were weighed separately and mixed together with a shovel prior to their introduction into the bioreactor. A poultry manure derived compost (PLC) was added at approximately  $10\% \pm 2\%$  on a wet weight basis to the OFMSW mixture to provide microbial seeding and to enhance the initiation of the composting process. The compost was derived from a large poultry breeding plant in Alexandroupolis (North Greece) that produces and markets poultry manure derived compost. Approximately 5 kg of tap water was added to each run to achieve an optimum mixture moisture content of  $49\% \pm 2\%$  ww. The wet weight of the simulated MSW that was used in each of the 6 runs was approximately 30 kg (ww) or 16 kg (dw). Hereon, the term “OFMSW” will refer to the mixtures of the OFMSW with the compost.

### 2.2. Household batteries

Eleven (11) types of zinc–carbon (ZC) and alkaline–manganese (AM) SHB were mixed with the OFMSW in 5 of the runs. Batteries, which were up to 3–4 months old, were collected from various recycling bins from the city of Xanthi (Greece). The ratio of alkaline: non-alkaline batteries was arbitrarily set at 4.3:1 (weight basis). This ratio was kept constant throughout all runs. All battery brands and types are mentioned in Table 1.

### 2.3. Experimental design

The experimental design involved 6 pilot scale runs with mixtures of the OFMSW and the SHB. One run was the control run (CNTR) that contained only the OFMSW without batteries, while the 5 other runs

**Table 1**  
Total metal content ( $\text{mg kg}^{-1}$  dw) of all materials.

Battery types and organic substrates	Weight per item (g)	Pb	Cd	Ni	Co	Cr	Cu	Fe	Zn	Mn
GP AA <sup>&amp;,*</sup>	16.9	$761 \pm 1\%$	$2.0 \pm 57\%$	$22.2 \pm 9\%$	$23.1 \pm 13\%$	$21.2 \pm 84\%$	$20.0 \pm 16\%$	$246,000 \pm 23\%$	$222,000 \pm 1\%$	$140,000 \pm 40\%$
Rotosonic AA <sup>*,&amp;</sup>	11.2	937	66.0	672	33.0	8.20	1040	31,980	176,100	86,600
UCAR AA <sup>*,&amp;</sup>	15.6	1400	0.441	139	13.0	18	199	41,600	251,000	151,000
Toshiba AA <sup>&amp;,*</sup>	17.2	$690 \pm 16\%$	$2.20 \pm 73\%$	$28.0 \pm 6\%$	$18.0 \pm 16\%$	$20.0 \pm 9\%$	$64.0 \pm 111\%$	$213,000 \pm 4\%$	$233,500 \pm 12\%$	$123,000 \pm 46\%$
Duracell Plus AA <sup>*,&amp;,&amp;</sup>	24.5	6.41	0.45	2230	7.40	9.30	11,850	166,000	195,500	157,000
Duracell plus AAA <sup>&amp;,&amp;,*</sup>	11.1	$24.3 \pm 26\%$	$1.8 \pm 21\%$	$3730 \pm 20\%$	$23 \pm 18\%$	$19 \pm 74\%$	$26,200 \pm 6\%$	$232,000 \pm 15\%$	$180,000 \pm 3\%$	$249,000 \pm 13\%$
UCAR Gold AA <sup>&amp;,&amp;,*</sup>	23.4	$7.42 \pm 5\%$	$0.83 \pm 76\%$	$1790 \pm 9\%$	$170 \pm 35\%$	$13 \pm 71\%$	$7160 \pm 10\%$	$236,000 \pm 23\%$	$185,000 \pm 6\%$	$150,000 \pm 41\%$
UCAR Gold C <sup>&amp;,&amp;,*</sup>	65.6	$5.08 \pm 27\%$	$0.39 \pm 9\%$	$984 \pm 10\%$	$6.1 \pm 3\%$	$5.4 \pm 18\%$	$1460 \pm 75\%$	$26,800 \pm 49\%$	$97,000 \pm 19\%$	$76,900 \pm 27\%$
Varta Energy AA <sup>&amp;,&amp;,*</sup>	23.6	$12.9 \pm 75\%$	$0.96 \pm 72\%$	$1320 \pm 71\%$	$242 \pm 66\%$	$11 \pm 77\%$	$12,800 \pm 12\%$	$141,000 \pm 26\%$	$170,000 \pm 19\%$	$139,000 \pm 61\%$
Aerocell AAA <sup>&amp;,&amp;,*</sup>	11.0	$20.2 \pm 25\%$	$2.2 \pm 54\%$	$2950 \pm 22\%$	$151 \pm 41\%$	$30 \pm 103\%$	$24,500 \pm 29\%$	$205,000 \pm 27\%$	$188,000 \pm 22\%$	$302,000 \pm 26\%$
Sunlight Max C <sup>*,&amp;,&amp;</sup>	67.7	2.80	0.710	1280	3.70	3.31	109	38,000	96,000	99,700
Food waste (FW) <sup>S</sup>		<LOD	<LOD	<LOD	<LOD	<LOD	$4.98 \pm 59\%$	$154 \pm 54\%$	$41.3 \pm 35\%$	$21.3 \pm 104\%$
Office paper (OFP) <sup>SS</sup>		$16.7 \pm 48\%$	$0.910 \pm 83\%$	$2.60 \pm 94\%$	$1.70 \pm 165\%$	<LOD	$8.18 \pm 73\%$	$1320 \pm 76\%$	$74.3 \pm 90\%$	$38.2 \pm 131\%$
Compost (PLC) <sup>SSS</sup>		$29.5 \pm 3\%$	$2.22 \pm 10\%$	$14.1 \pm 23\%$	$8.80 \pm 27\%$	<LOD	$56.7 \pm 12\%$	$1150 \pm 10\%$	$515 \pm 2\%$	$614 \pm 1\%$

LOD: level of detection.

\* Mean  $\pm$  coefficient of variation based on  $n=2$ .

\*\* One item was analyzed.

& Non-alkaline batteries.

&& Alkaline batteries.

<sup>S</sup>  $n=10$ .

<sup>SS</sup>  $n=7$ .

<sup>SSS</sup>  $n=2$ .

contained batteries at 3 concentration levels, namely: 0.98% (low level), 5.2% (medium level) and 10.6% (high level) (on wet OFMSW weight basis). The medium and high level contents are expected to be much higher compared to the actual contents of SHB in the Hellenic commingled MSW stream. In addition, commingled MSW are known to contain other metallic items, different to SHB that may affect the metal content of the OFMSW. Other metallic items were not included in the experiment, since the objective of the work was to solely investigate the effect of SHB to the OFMSW. Similar battery types and brands were used in all 5 runs.

Duplicate runs were performed at the medium and high levels in order to check the replicability of the experimental setup. Replication of the experimental setup was well established with the replicate runs at the medium and high levels, so as to render the use of additional replication at the control and low level unnecessary.

#### 2.4. Sampling process and analyses

Food wastes, office paper and the poultry derived compost were, each, characterized separately. Moisture contents were measured at 75 °C, till constant weight; the organic matter (OM) content was measured via the loss on ignition (LOI) at 550 °C for 2 h (USDA and USCC, 2002). During the intermediate and final sampling events, the entire contents of each bioreactor were removed and spread uniformly on a flat surface. Material was, then, quartered and two opposite quarters were randomly selected and placed back in the bioreactor. The remaining half was mixed again and randomly divided in 4 quarters again; the same procedure was followed until a representative amount of approximately 1.0 to 1.2 kg (ww) was reached. This amount was further mixed and divided into three sub-samples that were each characterized separately. Each sub-sample was, therefore, between 300 g and 400 g (ww), which was enough for characterization and metal analyses. The total dry mass of the OFMSW contained in each bioreactor at each sampling event was computed based on the principle of ash conservation. The sample size ( $n=3$ ) was considered optimal in this research. If a greater number of samples ( $n$ ) had been used during intermediate sampling events, the OFMSW to batteries ratio in the bioreactor would have been significantly affected (reduced), since batteries were never removed from the bioreactor during the intermediate sampling events.

After drying, grinding (with a porcelain mortar and a pestle) and homogenization of the OFMSW samples and the raw organic materials, approximately  $1.6 \text{ g} \pm 0.1 \text{ g}$  was randomly received from each material to analyze metals. One or two battery items, separately from each brand and type, were analyzed for their metal contents at the beginning of the experiments. At the end of the process, two (2) battery items from each type were randomly sampled, weighed, and analyzed for their total metal contents as well. Only in run 1, all battery items contained in the bioreactor at the end of the experiment were each separately analyzed.

The total “environmentally available” metal content of all organic materials (food wastes, office paper, compost and OFMSW) and of all 11 battery types was measured via an acid digestion technique according to method 04-12-B by USDA and USCC (2002), which is based on method 3050B (USEPA, 1996). The acid digestion technique that was adopted here is not a total digestion technique, but is a strong acid digestion that can dissolve almost all elements that can become “environmentally available” (USEPA, 1996). The goal of the work was, therefore, to quantify the metals that are mobile in the environment, since metals contained in the undissolved fraction after an acid digestion process can never become environmentally available. That is, the acid digestion procedure, as opposed to a total digestion technique, is expected to lead to the maximum battery dissolution that could take place under extreme environmental conditions.

The acid digestion procedure adopted is briefly described as follows: batteries were first cut in 3–4 pieces with a large scissor to facilitate acid digestion. All battery pieces were then placed into a Erlenmeyer flask and 20 ml of a 1:1  $\text{HNO}_3:\text{H}_2\text{O}$  solution was added. The solution was heated to around  $90 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$  and after 15 min, 10 ml of concentrated  $\text{HNO}_3$  was added. After 30 min of heating, 6 ml of condensed HCl was added into the beaker and heating continued for 1 h. Finally, 6 ml of  $\text{H}_2\text{O}_2$  was slowly added and the solution was mixed. Approximately (20) ml of deionized water was finally added to wash flask walls and heating was stopped after 10 min. After cooling, the solution was vacuum filtered through a  $0.45 \text{ }\mu\text{m}$  glass fiber filter. Filtrate was then placed into a 100 ml volumetric flask and deionized (DI) water was added to reach a final volume of 100 ml. The same procedure was followed to acid digest the organic materials. A blank, which contained no sample, was carried through the acid digestion procedure to establish a baseline for each metal. The undissolved fractions of some of the fresh batteries after acid digestion were quantified and were found to range from 5% to 30% of their initial dry weights. The total metal content was expressed on a per initial dry weight basis ( $\text{mg kg}^{-1} \text{ dw}$ ) for all materials used in this work.

With regard to the OFMSW, two intermediate and one final sample were received from each bioreactor. Batteries were not analyzed during the intermediate sampling events to avoid removing them from the bioreactor.

Nine heavy metals (Pb, Cd, Ni, Co, Cr, Cu, Fe, Zn and Mn) were then quantified using a flame atomic absorption spectrometer (FAAS) by Varian® model SpectrAA. Metal standards were prepared after dilutions of commercially available liquid solutions provided by MERCK® at initial concentrations equal to 1000 ppm. During FAAS analysis, a 4 point-calibration curve was prepared at the beginning of a series of measurements. A verification was performed every 10 sample measurements using one of the prepared standards. If deviation from the expected standard concentration exceeded 5%, a new calibration would always be performed. The limit of detection (LOD) was calculated for each metal as the concentration that would give a signal greater than 3.89 standard deviations above the mean noise level (based on  $n=7$  and at a 95% confidence level) according to APHA et al. (1998). The LODs were: 190 ppb for Pb, 10 ppb for Cd, 130 ppb for Ni, 100 ppb for Co, 300 ppb for Cr, 20 ppb for Cu, 170 ppb for Fe, 9 ppb for Zn and 40 ppb for Mn. All concentrations below the LOD were taken equal to 0 during statistical analysis.

Leachates produced at the intermediate and final sampling events were collected and removed through the bottom air inlet valve. The total leachate volume was measured and a sample of approximately 30 ml was stored for metal analyses using the same acid digestion procedure mentioned previously. The remaining leachate was recirculated back to the material in the bioreactor. The total mass of metals (mg) contained in the leachate collected at each sampling event was calculated based on the leachate volume (l) and the corresponding metal concentration ( $\text{mg L}^{-1}$ ).

Appropriate dilutions of the sample were performed when the original sample concentration exceeded the upper calibration point. The maximum dilutions attained during analyses of the batteries were: 1:20 for Pb, 1:10 for Cd, 1:500 for Ni, 1:100 for Co, 1:1 for Cr, 1:500 for Cu, 1:10000 for Fe, 1:75000 for Zn and 1:15000 for Mn. The maximum dilutions attained during the analysis of the OFMSW and its components were 1:30 (the highest dilutions were performed during the iron and zinc analyses in the high level runs). No dilutions were performed during leachate analyses.

#### 2.5. Bioreactor setup

The bioreactors were made of HDPE and had a total volume of approximately 230 l. They were insulated with a 5 cm thick styrofoam material and were kept at room temperature. Materials were placed on top of a stainless steel support screen to maintain a 15 cm air

plenum above the bottom of each bioreactor. Air was continuously pumped into the bottom of each bioreactor at a 2 l/min flowrate, using an air compressor. Temperature was manually recorded on a daily basis via a thermocouple, which was placed in the center of the waste material and was attached to a flexible plastic 2 m probe. A semi-airtight metallic cover, placed on top of each bioreactor, allowed the periodic recording of O<sub>2</sub>, CH<sub>4</sub> and CO<sub>2</sub> contents (in % v/v) at the headspace of the bioreactor above the waste surface. The gas concentration measurements were performed with a portable gas analyzer (GA 94, Geotechnical Instruments Ltd., UK). The runs lasted from 45 (run 1) to approximately 60 days (in all other runs). Run 1 was terminated earlier than the other runs (after 45 days as opposed to 60 days) in order to study potential differences in batteries' dry weight losses at the two different composting times. The bioreactor setup is shown in Fig. 1.

## 2.6. Calculations

The mere knowledge of the concentration (mg dry kg<sup>-1</sup>) of metals in the OFMSW would not provide any clear information on their potential transfer from the batteries to the OFMSW. This is because the concentration of metals in the OFMSW is, anyway, expected to increase during a composting process due to the reduction of dry matter. Some metals may be transferred to the leachate, if any. The total metal mass (solid and liquid form) is expected to remain constant within a bioreactor, since no volatilization of metals occurs. Therefore, if no transfer of metals from the batteries to the OFMSW occurs during the process, the mass of metals contained in the OFMSW should be maintained constant. In case of transfer, an increase of the metal mass in the OFMSW will occur concurrently with a likewise decrease of the batteries metal mass.

The mass of each metal in the OFMSW in the bioreactor, at variable times during the process, was computed according to Eq. (1):

$$\text{metal\_mass}_t = \frac{\text{Ash}_0}{1 - \text{VS}_t} \cdot \text{metal\_conc}_t \quad (1)$$

With: metal<sub>mass</sub><sub>t</sub> = the dry mass of a metal (mg) contained in the OFMSW at time t during the composting process; Ash<sub>0</sub>: the mass of the ash (kg) of OFMSW placed in the bioreactor at time 0; VS<sub>t</sub>: the volatile solids of the OFMSW at time t (expressed as a fraction on a dw basis) and metal<sub>conc</sub><sub>t</sub>: the total metal content (mg kg<sup>-1</sup> dw) of the OFMSW at time t.

Statistical comparisons (Dunnett's t-test) were performed using the masses of metals contained in the OFMSW to:

1. Investigate statistically significant variations during a run compared to time 0.
2. Investigate statistically significant variations in the runs containing batteries compared to the control at various times during the process.

A mass balance was performed to check whether potential increases in the mass of a metal in the OFMSW at the end of the process were accompanied by likewise decreases in the battery's metal mass.

## 3. Results and discussion

### 3.1. Initial characterization of materials

The FW, OFP and the PLC placed in the bioreactors had moisture contents equal to 66%, 4.8%, and 7.0% ww, respectively. All batteries had moisture contents between 0.8% and 0.9% ww. The organic matter

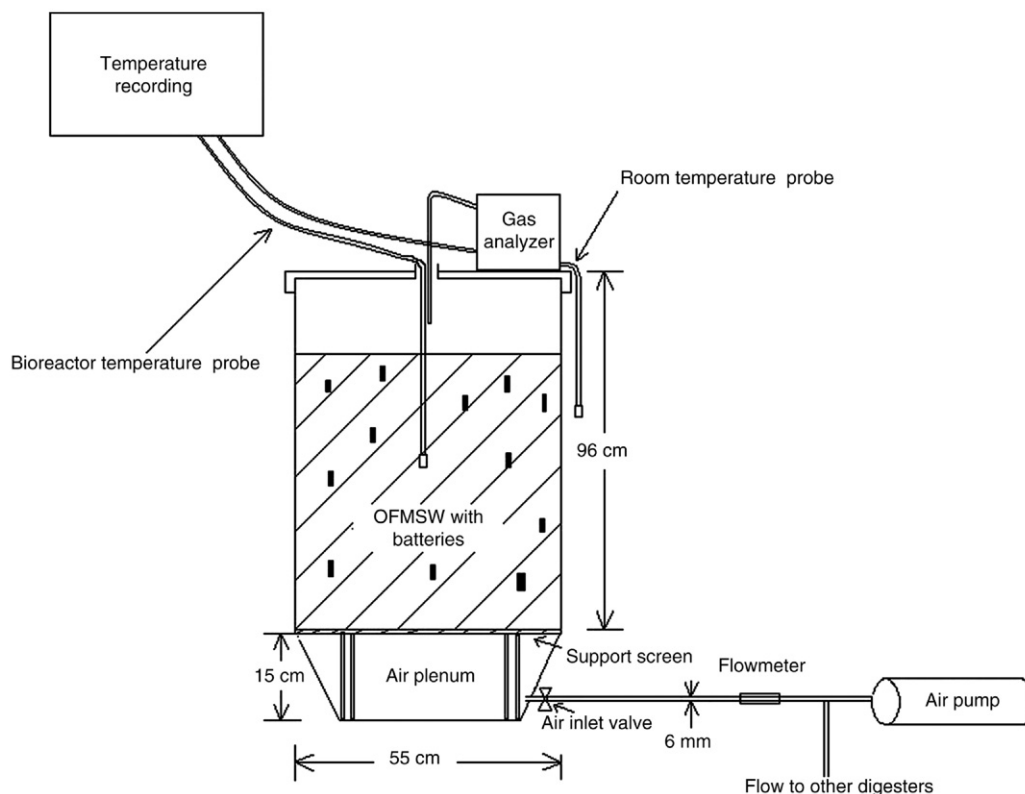


Fig. 1. Bioreactor setup (not to scale).



**Table 2**Mixture properties and total average initial metal contents (mg kg<sup>-1</sup> dw) achieved in the 6 runs.

Run	(1)	(2)	(3)	(4)	Pb	Cd	Ni	Co	Cr	Cu	Fe	Zn	Mn
Control	0	0.0	0	–	14	0.90	3.8	2.4	<LOD	16	841	141	136
Run 1	4 NALK and 8 ALK	0.30	0.98%	102: 1	16	0.97	24	3.3	4.1	103	2540	2360	2090
Run 2_1 Run 2_2	20 NALK and 60 ALK	1.55	5.2%	19: 1	29	1.0	142	6.0	4.7	824	12,900	14,900	12,400
Run 3_1 Run 3_2	40 NALK and 120 ALK	3.15	10.6%	9.5: 1	36	0.94	262	11	5.4	1510	27,700	28,900	23,800

The initial metal content is the average value based on the mixture that includes the batteries and the OFMSW.

(1): Battery items placed in the bioreactor; NALK: non-alkaline batteries and ALK: alkaline batteries.

(2): Total weight of batteries added (kg).

(3): Percentage of batteries added to the bioreactor expressed per wet weight of the OFMSW (as collected basis).

(4): Ratio of the wet OFMSW (as collected) to the (wet) weight of batteries included in the mixture.

LOD: level of detection.

contents of FW, OFP and the PLC were 93.1%, 80.3% and 53.5% dw, respectively. Table 1 includes the total heavy metal content of all 11 types of batteries used in this research as well as of the initial 3 organic substrates (i.e. food wastes, office paper and compost).

According to Table 1, Fe, Zn and Mn were the dominant metals in all types of batteries with contents up to approximately 300,000 mg dry kg<sup>-1</sup> in some battery types; the lowest content measured was approximately 27,000 mg dry kg<sup>-1</sup>. Cadmium was present in relatively small amounts (<5 mg dry kg<sup>-1</sup>) in almost all the batteries, except in one non-alkaline battery (Rotasonic AA) which had a cadmium content at 66 mg kg<sup>-1</sup> dw. The concentration of lead was very variable among batteries and ranged from 2.8 to 1400 mg kg<sup>-1</sup> dw. The non-alkaline batteries had a much higher lead content (from 690 to 1400 mg kg<sup>-1</sup> dw)—compared to the alkaline ones (with a corresponding range from 2.8 to 24 mg kg<sup>-1</sup> dw). On the other hand, nickel and copper were present in much higher concentrations in the alkaline batteries compared to the non-alkaline ones. Metals were also detected in the organic substrates, but in generally much lower concentrations compared to the batteries. For example, iron was found at a maximum concentration of approximately 1300 mg kg<sup>-1</sup> dw in OFP, zinc was found at a maximum concentration of approximately 500 mg kg<sup>-1</sup> dw in the compost and manganese was found at a maximum concentration of approximately 600 mg kg<sup>-1</sup> dw in the compost as well. Poultry compost contained a higher concentration of almost all metals compared to food wastes and office paper, except for iron. Iron was found at a higher concentration in FW and OFP compared to all other metals. Lead, cadmium nickel, cobalt, and chromium were not detected in the food wastes. Chromium was not detected in any of the OFMSW components.

The initial organic matter content of the OFMSW ranged from 79.9% to 82.5% (dw). The initial OFMSW weight placed to each of the 6 bioreactors (including the additional tap water to reach optimum moisture) was approximately 39 kg for all runs (see Table 2). The total weights and number of individual batteries added to each run are included in Table 2. Table 2, also, includes the initial averaged metal contents of the mixtures (i.e. OFMSW and batteries).

According to Table 2, the average initial contents of the 3 dominant metals in the mixtures (i.e. Fe, Zn and Mn) ranged from approximately 841, 141 and 136 mg kg<sup>-1</sup> dw, respectively, in the control (run w/o batteries) to approximately 27,700, 28,900 mg kg<sup>-1</sup> dw and 23,800 mg kg<sup>-1</sup> dw in the high level runs. Copper was the next dominant metal in the overall mixture (from 16 to 1500 mg kg<sup>-1</sup> dw) and the remaining five metals were present in average concentrations less than 300 mg kg<sup>-1</sup> dw.

### 3.2. Organic matter stabilization

The profile of the organic matter content of the OFMSW versus time is illustrated in Fig. 2a for all 6 runs. Apparently, organic matter (OM) degradation proceeded similarly for all runs regardless of the battery content. The final OM content at the end of the composting process was approximately 60% to 62% (dw) for all runs. Based on the principle of ash

conservation, the dry matter reductions of the OFMSW in the 6 runs at the end of the experiments were 47% (control), 49% (run 1), 47% (run 2\_1), 52% (run 2\_2), 50% (run 3\_1) and 56% (run 3\_2). There was a good replication of the organic matter reduction throughout the whole process for replicate runs 2\_1 and 2\_2. Run 3\_1 observed a slightly lower rate of OM reduction in the middle of the process compared to its replicate run 3\_2 (see Fig. 2a). Still, final OM reductions were statistically similar for runs 3\_1 and 3\_2 as well. The final moisture contents at the end of composting process were: 66% 50%, 65%, 49%, 48% and 62% (ww basis) for the control run and runs 1, 2\_1, 2\_2, 3\_1 and 3\_2, respectively. Therefore, moisture contents never reduced below the starting value during the process.

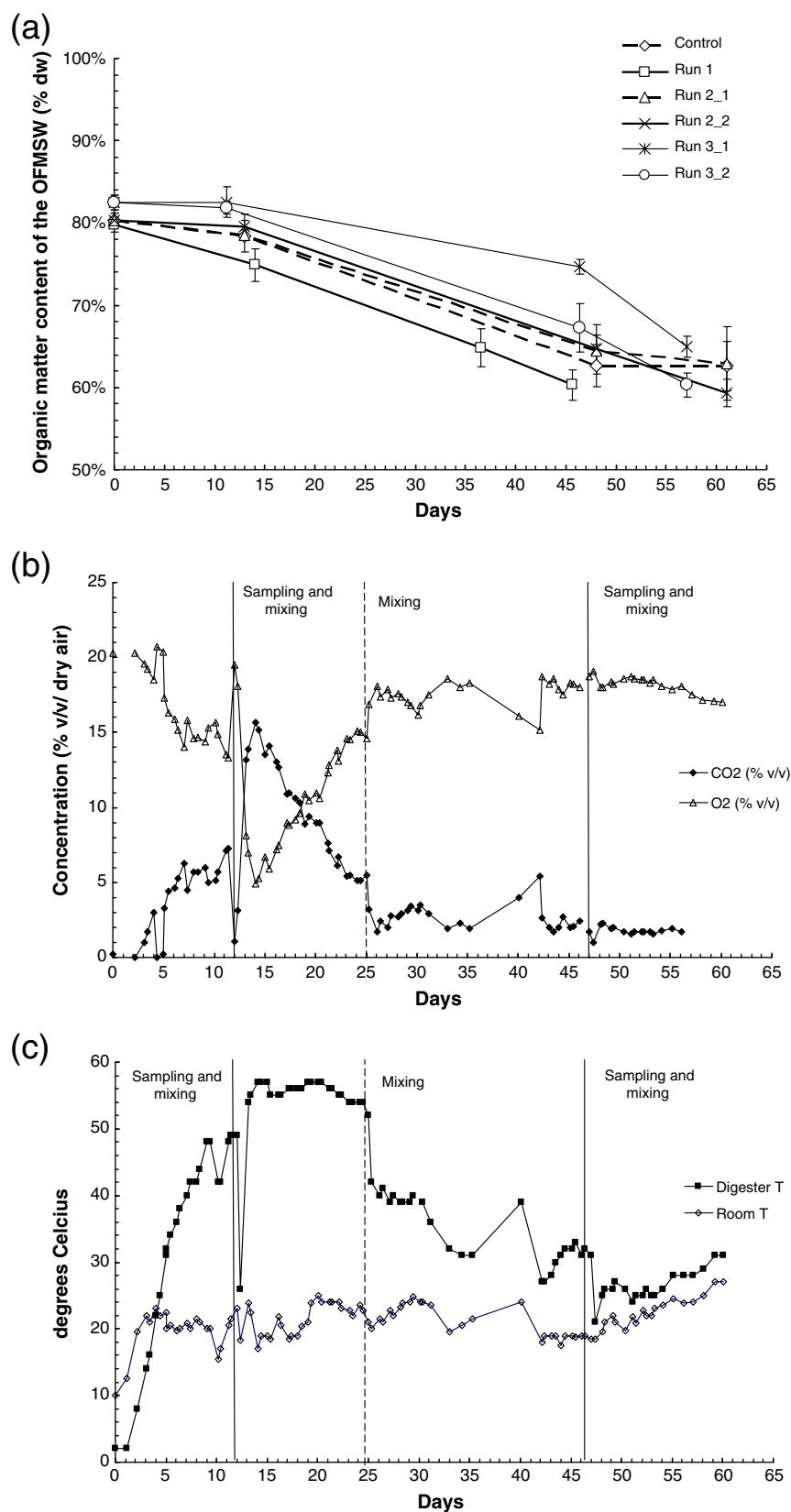
### 3.3. Gas concentration and temperature profiles

Fig. 2b illustrates the concentration of O<sub>2</sub> and CO<sub>2</sub> (% v/v) at the headspace of the bioreactor of the control run during the composting process. A gradual reduction of the O<sub>2</sub> concentration and a corresponding increase of the CO<sub>2</sub> concentration up to approximately day 15 were observed. The maximum aerobic activity was recorded between the 12th and 17th days from the start of all runs. There were temporary increases of microbial activity (increases in CO<sub>2</sub> contents and decreases of O<sub>2</sub> contents) right after the sampling and mixing events. The temperature profile of the control run is shown in Fig. 2c. Temperature followed a parallel profile to the CO<sub>2</sub> concentration profile. That is, a gradual temperature increase was observed up to approximately day 15. Then, thermophilic temperatures were maintained up to day 25, with a gradual decrease to room temperatures later on. Mixing and sampling events led to temporary temperature drops, but, eventually, aided the overall microbial activity. The gas concentration and temperature profiles of the control were typical for a composting process and similar to all the other runs; therefore, not all profiles are illustrated. Temperatures up to 65 °C were recorded for 3 of the runs, with 54 °C being the minimum of the peak temperatures recorded in all 6 runs. Conclusively, all runs followed the expected composting evolution profile.

### 3.4. Time profile of the metal masses in the OFMSW

Figs. 3–5 illustrate the mass profiles of 8 metals, contained in the OFMSW, versus time. Chromium is not included in the analysis, since its concentration was always below the LOD. According to the figures, the mass of all metals in the control remained stable during the process. For some metals, there was no clear trend versus time. Runs 2\_1 and 2\_2 had similar time trends for all metals, which is indicative of the good replication of the experimental setup. A good replication was also proven with runs 3\_1 and 3\_2 for almost all metals.

According to Fig. 3, lead, in run 1, showed a gradual increase after 15 days of composting. Rather sharp decreases and increases of the mass of lead were recorded for run 3\_1. Lead had similar profiles in all runs (except run 1). The mass of cadmium was similar for all runs at the end of the process. However, relatively low cadmium contents



**Fig. 2.** Progress of the composting process. (a) Organic matter content of the OFMSW (means  $\pm$  1 std), (b) gas concentration profiles in the headspace of the bioreactor of the control run and (c) temperature profile in the control run (the gas concentration and temperature profiles versus time of the control run were typical for all other runs).

were measured for runs 3\_1 and 3\_2 between the 5th and 45th days of composting. Nickel showed to clearly increase gradually with time in the high level runs (3\_1 and 3\_2), but not in the other runs. Still, there

was a large variance in the last nickel measurement at run 3\_2. Copper (see Fig. 4) had a steady mass profile versus the composting time for all runs, except in run 3\_2 at the end of the process.

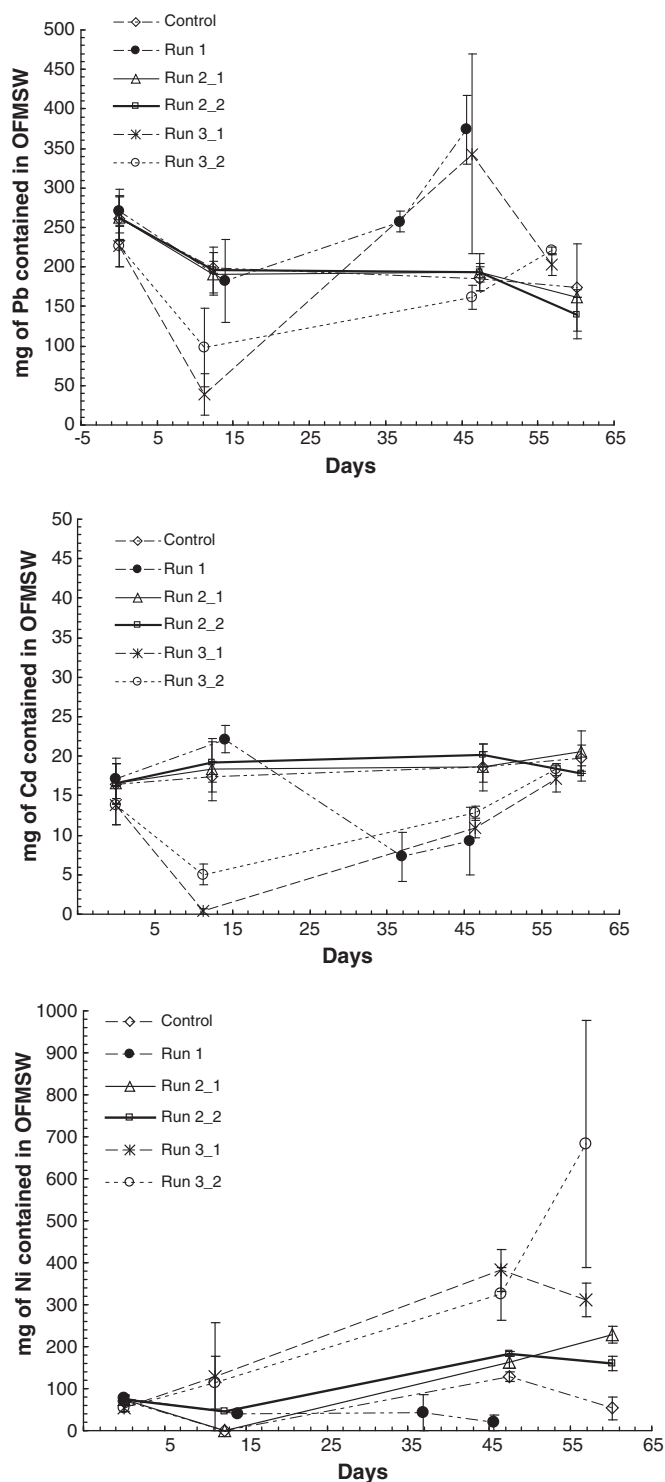


Fig. 3. Time profile of the masses of Pb, Cd and Ni, contained in the OFMSW (mean  $\pm$  std. error).

Zinc (Fig. 5) had a rather steady profile throughout time for all runs, except in run 3\_2 at which a large variance was calculated at the final sampling event. Iron, on the other hand, had a clear increasing trend with time in runs 3\_1 and 3\_2; this trend was not observed in runs 2\_1 and 2\_2. Manganese also showed a rather steady trend throughout the process for all 6 runs. Still, final values for manganese in runs 3\_1 and 3\_2 seemed to be lower compared to some of the other runs.

As is evident from Figs. 3 to 5, no clear conclusions can be drawn on potential differences in the masses of metals, except in the case of iron

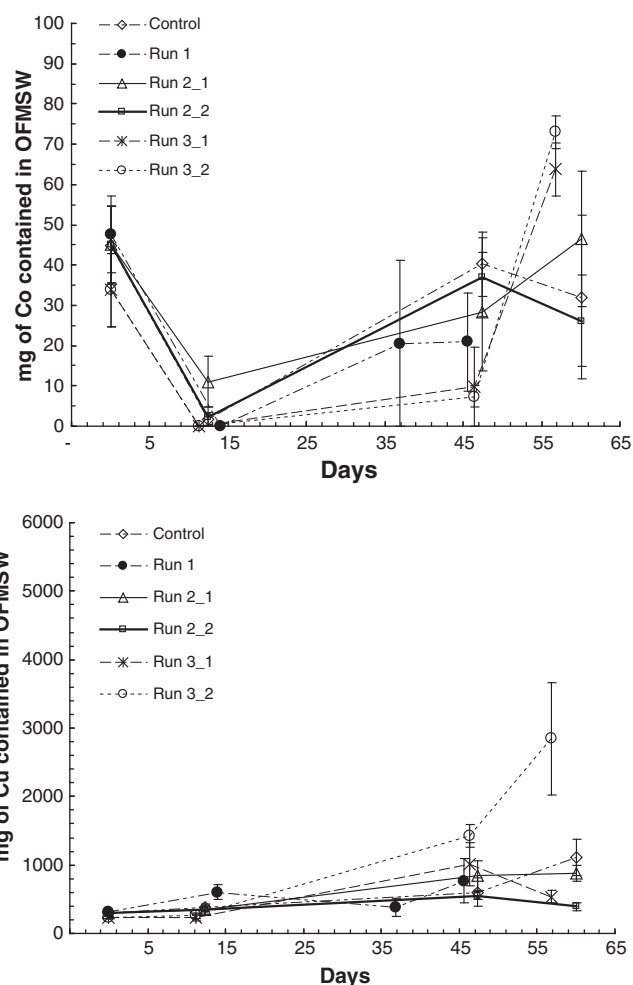


Fig. 4. Time profile of the masses of Co and Cu contained in the OFMSW (mean  $\pm$  std. error).

and nickel. For these 2 metals, there was a clear increasing trend with time in the high level runs. A statistical analysis (analysis of variance) was employed below to better investigate variations among metal masses.

#### 3.4.1. Statistical differences versus time

Although the graphical illustration of the data (Figs. 3–5) can aid in viewing potential trends, the statistical analysis amplifies the discussion of the results. Therefore, an analysis of variance (ANOVA) (Dunnett's t-test of comparisons with a control) was employed, according to Berthouex and Brown (2002). Dunnett's t-test calculated the confidence intervals (CI) of all potential differences of the means of the metal masses at the 3 sampling times (2 intermediate and 1 final) from the mean masses of the metals contained in the OFMSW at time 0 (time 0 was considered the "control" or "reference value"). Therefore, 3 differences were calculated for each metal in each run. The CIs of the differences of these mean values were obtained at a 95% joint confidence level. Table 3 includes all statistically significant differences (SSD).

According to Table 3, nickel showed a statistically significant increase (SSI) at the end of the process for runs 2\_1, 2\_2, 3\_1 and 3\_2 (compared to time 0). No such SSI was obtained at the low level run (run 1). This trend was evident in Fig. 2 too. Copper observed a statistically SSI in the control, which can be hardly explained and is rather attributed to sampling deficiencies. Still, copper had SSI with time in all runs, except run 1. Iron observed a SSI in runs 3\_1 and 3\_2. Particularly, in runs 3\_1 and 3\_2, iron showed to have SSI in

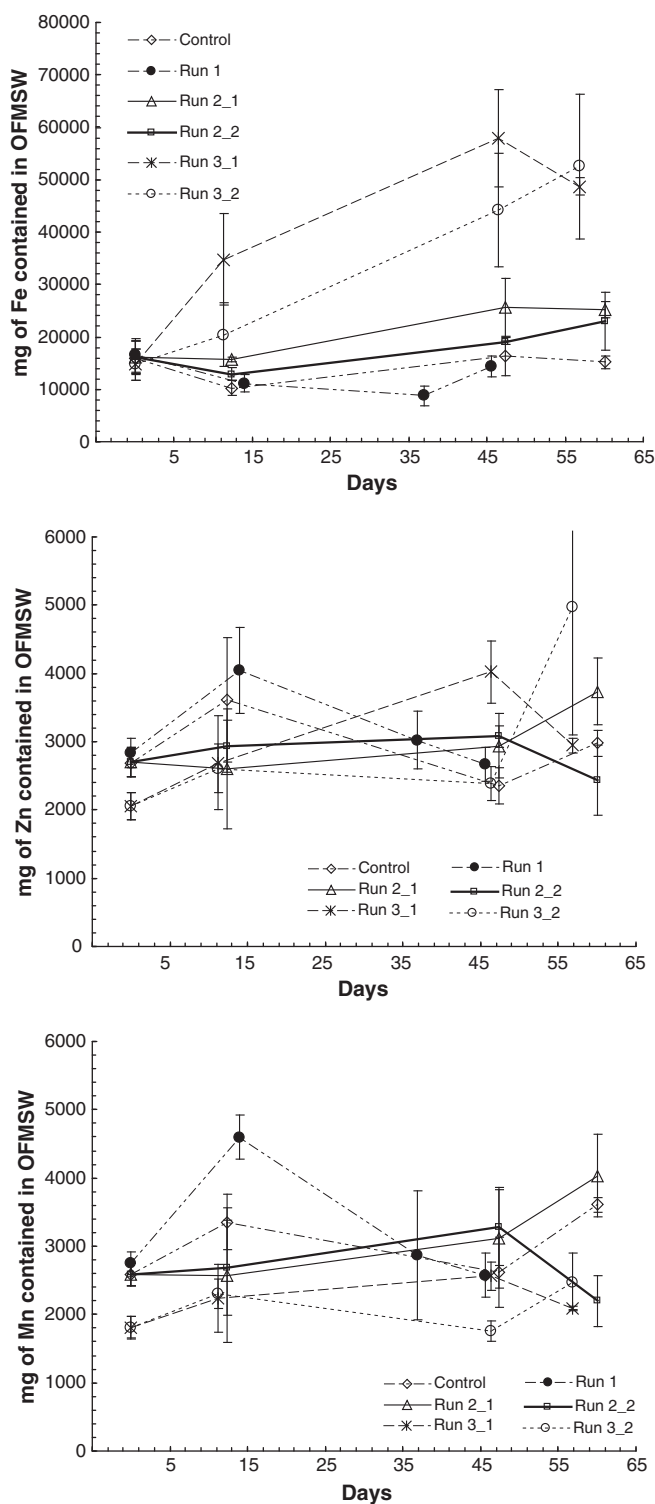


Fig. 5. Time profile of the masses of Fe, Zn and Mn contained in the OFMSW (mean  $\pm$  std. error).

intermediate times as well as at the end of the process. Zinc, in run 3\_2, also observed a SSI at the end of the process. A SSI was obtained for zinc on the 46th day of run 3\_1. Manganese, strangely, had a SSI only on day 14 for run 1, but no SSI was calculated in the other runs. No other SSI was obtained for manganese. Conclusively, the masses of Zn, Fe, Ni and Cu appeared to increase with time in the high level runs, which is an indication of metal transfer from the batteries to the OFMSW.

### 3.4.2. Statistical differences among runs compared to the control

Section 3.4.1 calculated the statistical differences during the composting time for each run separately. A question to be answered is whether the mass of a metal contained in the OFMSW of a run containing batteries differs from the corresponding metal mass in the control at the same sampling time. A transfer of metals to the OFMSW would be proven in case a SSI is actually obtained. The CIs of the differences of the means of the metal masses of runs 1 to 3 from the corresponding masses in the control, at the same sampling time, were calculated using Dunnett's t-test. Since, run 1 ended sooner than runs 2\_1, 2\_2, 3\_1 and 3\_2, it was not included in this analysis. Results are included in Table 4.

According to Table 4, there were few cases in which the runs that contained batteries showed a significantly lower mass of some metals compared to the control (for example, see Pb and Cd in the 1st intermediate sampling event). This contradiction is attributed to sampling deficiencies due to the inherent heterogeneity of MSW. Probably, if there were more replicates ( $n > 3$ ), such contradictions could have been alleviated. Still, sample size was limited by the overall mass of the material in the bioreactor, as mentioned earlier.

According to Table 4, nickel, copper and iron masses of run 3\_2 were significantly higher compared to the control's corresponding masses at the end of the process. Iron, in particular, showed a consistent SSI in run 3\_1 during the 1st, 2nd intermediate and final sampling events. That is, iron started to transfer from the batteries to the OFMSW during the first 12 days from the start of composting in a high level run. Nickel, also, appeared to significantly increase in runs 3\_1 and 3\_2 at the 2nd intermediate and final sampling times for run 3\_2. That is, nickel appeared to start to transfer to the OFMSW between the 12th and 46th days from the start of composting in the high level runs. Copper started to transfer to the OFMSW between the 46th and 57th days of composting in a high level run. No statistically significant differences were recorded for Pb, Cd, Co and Mn in any of the other runs. Zinc, in particular, showed a SSI in the 2nd intermediate sampling event of a high level run, but not in the final sampling event. From the above, it appears that nickel, copper and iron transferred from the batteries to the OFMSW in the high level runs, but not in the low and medium level runs.

### 3.5. Mass balance of metals

The total mass of each metal contained in all batteries at the start and end of a run was calculated by accounting for the average weight of a battery item, its average metal content and their number. According to the above, the dry weight reduction calculated for the alkaline batteries was 3% in run 1 and from 17% to 21% in runs 2\_1, 2\_2, 3\_1 and 3\_2. The dry weight reduction of the non-alkaline batteries never exceeded 3% in all runs. The overall combined dry weight reduction of the mixture of batteries was 2.5% in run 1 (that lasted 45 days) and from 14% to 17% for all the other runs (that lasted approximately 60 days). From the above, there are indications that a) the alkaline batteries were more prone to corrosion within the composting environment compared to the non-alkaline ones and b) the batteries' corrosion started to take place after the first 45 days of composting.

Fig. 6 illustrates the mass balances of 8 metals at each spike level. The metals' mass contained in the leachate was negligible compared to the metals' mass in the solid substrate contained in each bioreactor. Therefore, the former mass is not included in Fig. 6. The metals' mass balance closures are also included in Fig. 6 (percentages above bars). Closure was defined as the ratio (expressed in percentage) of the total metal mass (contained in the OFMSW + batteries) at the end of the process divided over the total metal mass (OFMSW + batteries) at the start of the process. According to Fig. 6, most balance closures were not far from the ideal value of 100%. Still, there were some extreme values (e.g. Cu in the control run with a closure equal to 371%). This extreme



**Table 3**

Statistical differences of the metals in the OFMSW compared to time 0.

Run	Pb	Cd	Ni	Co	Cu	Fe	Zn	Mn
Control	None	None	m <sub>47</sub> >m <sub>0</sub>	None	m <sub>60</sub> >m <sub>0</sub>	None	None	None
Run 1	None	None	None	m <sub>14</sub> <m <sub>0</sub>	None	None	None	m <sub>14</sub> >m <sub>0</sub>
Run 2_1	None	None	m <sub>47</sub> >m <sub>0</sub>	None	m <sub>47</sub> >m <sub>0</sub>	None	None	None
			m <sub>60</sub> >m <sub>0</sub>		m <sub>60</sub> >m <sub>0</sub>			
Run 2_2	m <sub>60</sub> <m <sub>0</sub> *	None	m <sub>47</sub> >m <sub>0</sub>	m <sub>12</sub> <m <sub>0</sub>	m <sub>47</sub> >m <sub>0</sub>	None	None	None
			m <sub>60</sub> >m <sub>0</sub>					
Run 3_1	None	m <sub>11</sub> <m <sub>0</sub>	m <sub>47</sub> >m <sub>0</sub>	None	m <sub>46</sub> >m <sub>0</sub>	m <sub>11</sub> >m <sub>0</sub>	m <sub>46</sub> >m <sub>0</sub>	None
			m <sub>60</sub> >m <sub>0</sub>			m <sub>46</sub> >m <sub>0</sub>		
						m <sub>57</sub> >m <sub>0</sub>		
Run 3_2	None	None	m <sub>60</sub> >m <sub>0</sub>	m <sub>57</sub> >m <sub>0</sub>	m <sub>57</sub> >m <sub>0</sub>	m <sub>46</sub> >m <sub>0</sub>	m <sub>57</sub> >m <sub>0</sub>	None
						m <sub>57</sub> >m <sub>0</sub>		

Dunnett's t-test was employed at a 95% joint confidence level (Berthouex and Brown, 2002). Comparisons were performed between the mass of metal in the OFMSW at each sampling event with the initial mass of metals at time 0. Based on Dunnett's test, the differences of the mean masses of the metals at the intermediate and final sampling events from the mean initial metal masses were calculated for each metal and for each run, separately. Therefore, 3 confidence intervals were calculated per metal and per run at a 95% joint confidence level.

\* Subscript indicates the time (at days) of the sampling event and m indicates the mass (mg) of the corresponding metal contained in the OFMSW in the bioreactor. The "<" or ">" symbols indicate statistically significant differences at a 95% joint confidence level. For example, the Zn mass in the OFMSW of run 3\_2 on day 57 (end of process) was significantly higher compared to the Zn mass at the start of the experiment (time 0).

value can be explained by the fact that copper was actually in very low amounts in the control run. If the above extreme value is excluded, then average closures calculated for each metal separately from all 6 runs are: 113% for lead, 114% for cadmium, 110% for nickel, 82% for cobalt, 110% for copper, 115% for iron, 107% for zinc and 113% for manganese.

On the other hand, it is unclear from Fig. 6 whether a decrease in the metal content of the batteries corresponded to the same increase in the OFMSW. This was attributed to the large variances as well as to the fact that the amounts of battery related metals were much larger compared to OFMSW related metals. In some cases, the (average) mass of metals contained in the batteries at the end of the process was greater than the (average) mass of the same batteries at the start of the run (e.g. see Ni, Fe, Zn and Mn in runs 2\_1 and 2\_2). Based on visual inspection, almost all batteries at the end of the 60-day runs 2 and 3 were actually rusted (as opposed to the batteries in run 1 that lasted 45 days).

### 3.6. Leachate quality

Although leachate is not a major concern in MSW composting facilities, the leachates generated at each run were collected and analyzed for their total metal contents. The leachate volumes

extracted from all runs during all sampling events ranged from 1 l to 4 l. Copper, iron, zinc, and manganese were measured at concentrations above the LOD at all sampling events for all runs. On the other hand, lead, cadmium and nickel were detected at some sampling events and for some of the runs, as shown in Fig. 7. Fig. 7 presents metal concentrations in the leachate of all runs during all sampling events (note the log scale at the y axis). According to Fig. 7, the metals measured in the highest concentrations were copper, iron, zinc and manganese, as had been found by Karnchanawong and Limpitprakan (2009) and Xará et al. (2009) too. This is an expected finding, since the above 4 metals were the dominant metals in the whole system.

The concentration of almost all metals was kept below approximately 10 mg L<sup>-1</sup>, except in the case of iron, for which two relatively high values of 240 and 180 mg L<sup>-1</sup> were measured in the high level runs (i.e. runs 3\_1 and 3\_2, respectively). The concentration of copper and iron appeared to increase with composting time, while zinc and manganese had approximately constant concentrations during the process for all runs. The control had the lowest concentration of the 4 dominant metals (Zn, Mn, Fe and Cu) as expected. In addition, as the battery content increased within the bioreactors, the overall metals' concentration increased as well. Lead was below the LOD for the low and medium level runs. However, at the high level runs, lead concentration was quantifiable and never exceeded 10 mg l<sup>-1</sup>. Nickel, also, started to appear in the leachate in the medium and high level runs in concentrations that never exceeded 3.0 mg l<sup>-1</sup>. Cobalt and chromium were always below the LOD at all runs.

The metal concentrations measured in this work were compared against the leaching criteria set by European Decision 2003/33/EC (EC, 2003). This EU decision designates the criteria for the acceptance of wastes in various landfill types (inert, non-hazardous and hazardous) based on a specified test procedure using indicators such as certain heavy metals and other parameters. Although the test procedure adopted by the EU decision was not followed here, it would still be interesting to compare results with the limits. Therefore, according to the limits for Pb, Cu, Zn, Ni and Cd mentioned in the decision for non-hazardous landfills (respectively equal to 3, 30, 15, 3 and 0.3 mg l<sup>-1</sup>), the MSW at the end of these composting experiments could be directed to a non-hazardous waste landfill.

### 3.7. End product quality

There are many indicators to judge the quality of composted end products; one is the total metal content. The total metal contents of the end products (i.e. OFMSW) of all runs are included in Table 5 and are compared to the Greek limits for MSW derived composts (HMD,

**Table 4**

Statistical differences of the masses of 8 metals compared to the control.

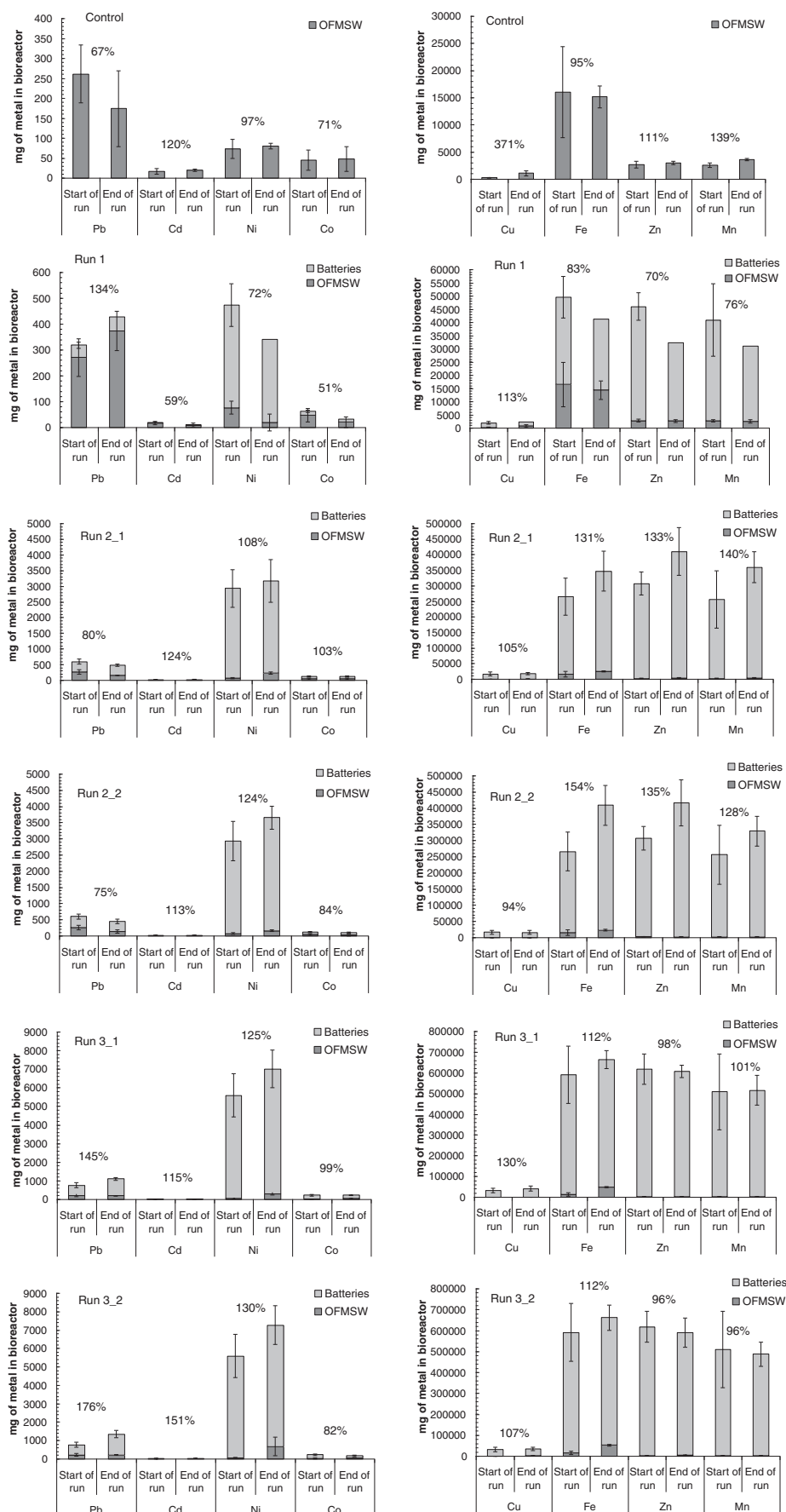
Metal	1st intermediate sampling*	2nd intermediate sampling**	Final sampling***
Pb	Run 3_1<Control	None	None
Cd	Run 3_1<Control	None	None
	Run 3_2<Control		
Ni	None	Run 3_1>Control	Run 3_2>control
		Run 3_2>Control	
Co	None	None	None
Cu	None	None	Run 3_2>control
Fe	Run 3_1>control	Run 3_1>control	Run 3_1>control
			Run 3_2>control
Zn	None	Run 3_1>control	None
Mn	None	None	None

Dunnett's t-test was employed at a 95% joint confidence level (Berthouex and Brown, 2002). The "<" or ">" symbols indicate statistically significant differences at a 95% joint confidence level.

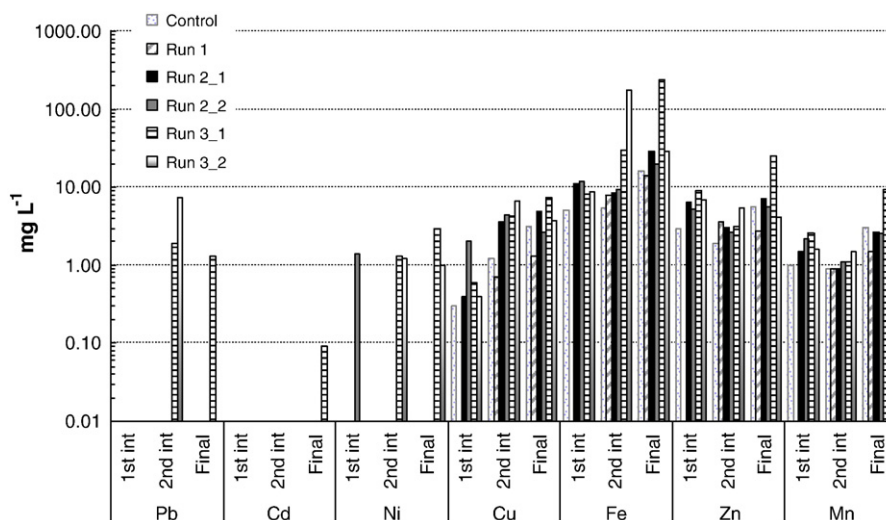
\* The 1st intermediate sampling took place on day 11 for the control run and runs 2\_1 and 2\_2 and on day 12 for runs 3\_1 and 3\_2.

\*\* The 2nd intermediate sampling took place on day 47 for the control run and runs 2\_1 and 2\_2 and on day 46 for runs 3\_1 and 3\_2.

\*\*\* Final sampling took place on day 60 for the control run and runs 2\_1 and 2\_2 and on day 57 for runs 3\_1 and 3\_2.



**Fig. 6.** Balance of metals (means  $\pm$  pooled std.dev). Percentages above bars denote the mass balance closures (closure is the ratio of the mass of a metal contained in the bioreactor at the end of the process over the mass of a metal contained in the bioreactor at the start of the experiment).



**Fig. 7.** Concentration of 7 metals in the leachates that were generated during composting. Zero values indicate concentrations below the LOD. (Cobalt and chromium are not illustrated since they were both below the LOD at all sampling events; "int": intermediate sampling event and "Final": final sampling event).

**Table 5**

Hellenic legislation limits and final metal contents of the OFMSW end product ( $\text{mg kg}^{-1}$  dw).

	(1)	Control	Run 1	Run 2_1	Run 2_2	Run 3_1	Run 3_2
Pb	500	$17 \pm 51\%$	$38 \pm 21\%$	$16 \pm 12\%$	$15 \pm 39\%$	$22 \pm 12\%$	$27 \pm 3\%$
Cd	10	$2.0 \pm 10\%$	$0.9 \pm 76\%$	$2.0 \pm 10\%$	$1.9 \pm 12\%$	$1.9 \pm 16\%$	$2.3 \pm 5\%$
Ni	200	$7.1 \pm 31\%$	$4.3 \pm 39\%$	$22 \pm 3\%$	$17 \pm 17\%$	$34 \pm 22\%$	$83 \pm 71\%$
Co	nl	$3.0 \pm 109\%$	$2.2 \pm 105\%$	$4.4 \pm 58\%$	$2.8 \pm 74\%$	$7.0 \pm 19\%$	$9.1 \pm 11\%$
Cr	510	<LOD	<LOD	<LOD	<LOD	<LOD	<LOD
Cu	500	$113 \pm 48\%$	$77 \pm 69\%$	$88 \pm 32\%$	$42 \pm 25\%$	$59 \pm 28\%$	$349 \pm 46\%$
Fe	nl	$1510 \pm 11\%$	$1470 \pm 26\%$	$2500 \pm 20\%$	$2450 \pm 38\%$	$5330 \pm 5\%$	$6450 \pm 42\%$
Zn	2000	$296 \pm 8\%$	$270 \pm 11\%$	$363 \pm 14\%$	$265 \pm 40\%$	$323 \pm 8\%$	$607 \pm 61\%$
Mn	nl	$360 \pm 4\%$	$261 \pm 17\%$	$391 \pm 18\%$	$239 \pm 32\%$	$230 \pm 3\%$	$308 \pm 32\%$

Mean  $\pm$  coefficient of variation ( $n = 3$ ).

(1): Limits of total metal content ( $\text{mg kg}^{-1}$  dw) for MSW derived composts according to the Hellenic legislation (HMD, 1997).

nl: no limit is mentioned.

1997). In all cases, the total metal contents were far below the legislated limits. Only copper, in run 3\_2, was close, but still lower than, the suggested limit of  $500 \text{ mg kg}^{-1}$  dw. Despite the above, the final end product from all runs can hardly be defined as compost, since no curing took place. The end material was simply the product of a 60 day active composting process without curing. As mentioned earlier, this research project was designed on the notion that the batteries present in the MSW stream after an active composting process will be removed, via screening, prior to curing.

#### 4. Conclusions

Conclusions from this work are:

- 1) The batteries' dry matter reduction was 6 to 7 times greater after a 60 day composting period compared to a 45 day composting period. Therefore, it appears that the structure of SHB starts to change after 45 days within an active composting environment.
- 2) Alkaline batteries lost from 17% to 21% of their initial dry mass during the 60-day composting process. On the other hand, the non-alkaline batteries lost from 0% to 3% of their dry mass during the composting process.
- 3) No transfer of metals from the batteries to the OFMSW was observed for the low and medium level runs. On the other hand,

nickel, iron and copper appeared to transfer from the batteries to the OFMSW in the high level runs.

- 4) There was a high internal variation among replicate metal measurements in some of the sampling events. On the other hand, there was a good replication of the overall experimental setup, as was judged by the results of replicate runs 2\_1, 2\_2 and replicate runs 3\_1 and 3\_2.
- 5) Metal mass balance closures ranged from approximately 51% to 176%. An extreme closure value (371%) was recorded for copper in the control run. It was not easy to discern a change in the metal mass of the OFMSW during the process and a similar opposite change in the batteries' metal masses.
- 6) The concentration of metals in the leachates was always less than  $10 \text{ mg l}^{-1}$  for most metals in all runs except iron that reached concentrations above  $150 \text{ mg l}^{-1}$  in the 2 high level runs. Chromium and cobalt were below the limits of detection in all runs.
- 7) Metal contents of the composted end products were below the Hellenic limits.

#### Acknowledgments

The authors wish to thank Professor E.A.Voudrias for his support throughout this project. The authors also thank Dr. Spyridon Ntougias and Dr. Ilias Zafiriadis for their useful comments on the manuscript.

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